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THE PRODUCTION OF ALLOYS OF TUNGSTEN AND OF MOLYBDENUM IN THE ELECTRIC FURNACE.[1]

BY CHARLES L. SARGENT.

Received September 17, 1900.

INTRODUCTION.

WITH the introduction of the electric furnace, the new field of chemistry at high temperatures was opened to investigation. Moissan especially succeeded in reducing the most refractory oxides, and in volatilizing many of them, as well as certain metals which were considered infusible. He described[2] a method of preparing alloys of vanadium, using the pentoxide as the source of vanadium.

Bernoulli[3] prepared alloys of tungsten with copper, lead, bismuth, cobalt, nickel, etc. His method of working consisted in mixing the oxides of the two metals with lampblack, placing the mixture in a crucible and then heating it in an ordinary furnace. Proceeding in this manner he was not able to obtain alloys containing more than 10 per cent. of tungsten.

[1] From author's thesis presented for the degree of Doctor of Philosophy, University of Pennsylvania, 1900.

[2] "La Four Electrique," p. 246.

[3] Pogg. *Ann.*, III, 573.

Knowing the comparative ease with which tungsten and molybdenum are reduced from their oxides, it was thought that it might be possible to mix these oxides with oxides of other metals and then reduce the mixture in the electric furnace, obtaining as a final product an alloy of the two metals.

In the following experiments the furnace used was of the type known as the "Moissan." The carbon for the reduction was prepared by ignition of cane-sugar. The crucibles were made of graphite, modeled in the form of an assay scorifier. This shape is preferable, as it allows the arc to play directly on the bottom of the crucible. In some of the experiments the crucibles were lined with magnesia, thus preventing the hot metal from absorbing any of the graphite.

Unless otherwise mentioned the graphite crucibles were used. After each reduction the furnace was closed and allowed to cool before removing the crucible containing the fusion.

Ten preliminary experiments were made with tungstic acid, attention being paid more particularly to the influence of the carbon, and to the variation in voltage and amperage. The results showed that the amount of carbon used exerted a greater influence in the purity of the metal than variations in the strength of current, and the duration of its action. The product invariably contained carbon and traces of unreduced oxide. An effort was made to burn out the carbon by heating the impure metal in magnesia-lined crucibles, but the metal was changed to trioxide and magnesium was volatilized.

ALLOYS OF TUNGSTEN.

Tungsten trioxide and bismuthic oxide were mixed in varying amounts with carbon and exposed for several minutes to the action of a current of 60 to 80 volts, and 75 to 150 amperes. The quantity of bismuth detected in the metallic product in no case exceeded 0.64 per cent.

When cupric oxide was substituted for the bismuthic oxide, and a current of 75 to 150 amperes and 60 to 80 volts was applied for five minutes a regulus remained which showed, upon analysis, 18.24 per cent. of tungsten, 77.73 per cent. of copper, and 3.23 per cent. of carbon. On using a crucible lined with magnesia the copper was completely expelled and tungsten trioxide remained.

The results obtained in attempting to prepare an alloy of tungsten and manganese were negative. Two trials were made with chromium with the following conditions:

Experiment I.—

2 grams of tungsten trioxide	Voltage, 72–80.
2 " " chromic oxide	Amperage, 100–160.
2 " " carbon	Time, 5 minutes.

The reduction was made in a lime crucible. The resulting globule of metal was very hard and brittle, its surface being covered with a layer of chromic oxide. Its interior was gray in color. The specific gravity of the alloy equaled 8.96. It showed, upon analysis, 2.87 per cent. of chromium and 97.64 per cent. of tungsten.

Experiment II.—

2 grams of tungsten trioxide	Voltage, 72–85.
2 " " carbon	Amperage, 100–150.
1 gram of chromic oxide	Time, 5 minutes.

A carbon crucible was used in this experiment, but alloys containing a higher percentage of chromium than given in Experiment I, were not prepared. The next trial was made upon tungsten trioxide and cobaltic oxide with these conditions:

2 grams of tungsten trioxide	Voltage, 70–80.
2 " " cobaltic oxide	Amperage, 125–150.
1 gram of carbon	Time, 1 minute.

The crucible was of lime and the button, which was strongly magnetic, very tough and tenacious, had the specific gravity 10.96. Its analysis showed 51.86 per cent. of tungsten, and 48.26 per cent. of cobalt.

Upon varying the conditions to

1 gram of tungsten trioxide	Voltage, 65–70,
3 grams of cobaltic oxide	Amperage, 110–130,
2 grams of carbon	Time, 2 minutes,

and using, as before, a lime crucible the resulting metallic globule was discovered to be strongly magnetic, very tough, and it was broken with difficulty. Its specific gravity was found to be 8.92. Its analysis revealed the presence of 29.24 per cent. of tungsten, and 70.10 per cent. of cobalt.

The experiments conducted with the oxides of tungsten and nickel gave very favorable results:

Experiment I.

2 grams of tungsten trioxide	Voltage, 68–80.
2 " " nickel oxide	Amperage, 100–135.
2 " " carbon	Time, 2 minutes.

The reduction was made in a crucible lined with magnesia, and a globule was obtained which was slightly magnetic, could be filed, and was very tough. Its specific gravity equaled 10.66. Its analysis gave 50.22 per cent. of tungsten, and 49.88 per cent. of nickel.

Experiment II.—

3 grams of tungsten trioxide	Voltage, 75–90.
2 " " carbon	Amperage, 100–175.
1 gram of nickel oxide	Time, 1½ minutes.

The crucible was similar to that used in Experiment I. The resulting button was very hard and non-magnetic, extremely brittle, and easily pulverized. Its specific gravity equaled 12.66. The analysis showed 91.19 per cent. of tungsten, and 8.08 per cent. of nickel.

Repeated attempts were made to alloy tin and tungsten after the plan pursued with the other metals, but without success. It would, therefore, appear that starting with the oxides it is possible to prepare alloys of tungsten with those metals which require a high temperature for their volatilization. In the case of those requiring a low temperature, the oxide is apparently reduced and the metal driven off before the tungstic oxide is reduced. Doubtless alloys of tungsten and the lower fusing metals could be prepared by starting with the metals and melting them together; but if this is attempted in the electric furnace the intense heat drives off the lower-fusing metal, leaving the one with a higher fusing-point in the furnace.

ALLOYS OF MOLYBDENUM.

Three reductions of bismuthic oxide and tungsten trioxide were made:

Experiment I.—

4 grams of molybdenum trioxide	Voltage, 65–70.
4 " " bismuth oxide	Amperage, 90–120.
1 gram of carbon	Time, 2 minutes.

The resulting metal was very hard. Its specific gravity equaled 6.81. It gave, upon analysis, 91.61 per cent. of molybdenum, 6.50 per cent. of bismuth, and 2.28 per cent. of carbon.

Experiment II.—

2 grams of molybdenum trioxide	Voltage, 60–70.
8 " " bismuth oxide	Amperage, 75–110.
1.5 " " carbon	Time, 2 minutes.

A granular mass was obtained after heating for one and one-half minutes. It was re-fused for one minute in a crucible lined with magnesia, and was converted into a very hard metallic button, having a specific gravity of 8.91. It showed, upon analysis, 92 per cent. of molybdenum, 4.81 per cent. of bismuth, and 3.90 per cent. of carbon.

Experiment III.—

6 grams of molybdenum trioxide	Voltage, 70–80.
2 " " bismuth oxide	Amperage, 100–150.
1 gram of carbon	Time, 2 minutes.

A crucible with magnesia lining was used in this experiment. The analysis showed 97.91 per cent. of molybdenum, 1.10 per cent. of bismuth, and 1.21 per cent. of carbon. All efforts looking to the formation of a molybdenum-copper alloy were fruitless.

In the case of molybdenum and manganese the results were good and entirely unlike those observed with tungsten and manganese, as will be noticed in the following experiments:

Experiment I.—

4 grams of molybdenum trioxide	Voltage, 75–85.
4 " " manganese dioxide	Amperage, 100–150.
3 " " carbon	Time, 2 minutes.

A button, having a specific gravity of 7.08, was obtained. It gave, upon analysis, 71.07 per cent. of molybdenum, 14.36 per cent. of manganese, 9.60 per cent. of iron, and 4.34 per cent. of carbon. The iron in this alloy came from the manganese dioxide.

Experiment II.—

2 grams of molybdenum trioxide	Voltage, 70–80.
6 " " manganese dioxide	Amperage, 100–130.
3 " " carbon	Time, 2 minutes.

The resulting alloy had the specific gravity 6.9. Its analysis

revealed the presence of 60.08 per cent. of molybdenum, 21.11 per cent. of manganese, 16.64 per cent. of iron, and 2.99 per cent. of carbon. My experience with molybdenum and chromium is briefly summarized in the following experiments:

Experiment I.—

2 grams of molybdenum trioxide	Voltage, 70–90.
6 " " chromic oxide	Amperage, 100–150.
2 " " carbon	Time, 3 minutes.

A carbon crucible was used in the reduction. Molybdenum trioxide, unmixed with carbon, was placed in the bottom of the crucible, and upon this trioxide was introduced a mixture of chromic oxide, the balance of the oxide of molybdenum and carbon. The product of the fusion was hard and brittle. Its specific gravity was found to be 6.53. Its analysis showed the presence of 12.82 per cent. of molybdenum, 76.71 per cent. of chromium, 7.52 per cent. of iron, and 2.55 per cent. of carbon.

Experiment II.—

4 grams of molybdenum trioxide	Voltage, 70–80.
4 " " chromic oxide	Amperage, 90–130.
3 " " carbon	Time, 3 minutes.

The alloy, steel-gray in color, proved to be hard and brittle. Its specific gravity was found to be 7.65, and upon analysis it showed 39.96 per cent. of molybdenum, 53.24 per cent. of chromium, and 6.22 per cent. of iron with a trace of carbon. An alloy of tin and molybdenum was not obtained.

Several trials were made with molybdenum and nickel oxides, but only those will be introduced here which gave definite results:

Experiment I.—

2 grams of molybdenum trioxide	Voltage, 70–80.
4 " " nickel oxide	Amperage, 75–125.
2 " " carbon	Time, 2 minutes.

The crucible was lined with magnesia for this fusion. The metal obtained was very hard and brittle, non-magnetic, and had a specific gravity of 7.61. Upon analysis it gave 17.72 per cent. of molybdenum, 80.93 per cent. of nickel, and 1.63 per cent. of carbon.

Experiment II.—

4 grams of molybdenum trioxide	Voltage, 75–80.
2 " " nickel oxide	Amperage, 100–150.
2 " " carbon	Time, 1 minute.

The alloy was soft and easily filed. It was non-magnetic and its specific gravity was found to be 8.00. Its analysis showed 65.10 per cent. of molybdenum, and 34.72 per cent. of nickel.

Experiment III.—

4 grams of molybdenum trioxide	Voltage, 73–75.
2 " " nickel oxide	Amperage, 140–160.
2 " " carbon	Time, 1 minute.

A graphite crucible was used in the reduction. The alloy was hard, brittle, and non-magnetic. Its specific gravity equaled 8.88. Its analysis showed the presence of 50.20 per cent. of nickel, 42.48 per cent. of molybdenum, 3.05 per cent. of carbon, and 4.04 per cent. of silica. The silica probably came from the material used in the manufacture of the graphite crucible.

The reduction of mixed oxides of molybdenum and cobalt was not attended with the least difficulty; indeed, from the appended results it would seem that these particular metals alloy in almost any proportion:

Experiment I.—

2 grams of molybdenum trioxide	Voltage, 68–75.
4 " " cobaltic oxide	Amperage, 100–175.
2 " " carbon	Time, 1½ minutes.

The crucible had a magnesia lining. The alloy was very hard, tough, and magnetic. Its specific gravity equaled 7.32. Its analysis gave 17.06 per cent. of molybdenum, and 82.34 per cent. of cobalt.

Experiment II.—

3 grams of molybdenum trioxide	Voltage, 70–90.
3 " " cobaltic oxide	Amperage, 100–160.
2 " " carbon	Time, 1½ minutes.

The crucible was lined with magnesia. The alloy was very hard, tough, and magnetic. Its specific gravity equaled 6.44. Its analysis showed 35.64 per cent. of molybdenum, 62.91 per cent. of cobalt, and 1.79 per cent. of carbon.

Experiment III.—

3 grams of molybdenum trioxide	Voltage, 75–80.
3 " " cobaltic oxide	Amperage, 175–190.
2 " " carbon	Time, 1½ minutes.

In this experiment I used a graphite crucible, and obtained an alloy that was hard, slightly magnetic, very brittle, and easily pulverized. The specific gravity was 6.94. Its analysis gave 47.10 per cent. of molybdenum, and 52.30 per cent. of cobalt.

Experiment IV.—

4 grams of molybdenum trioxide	Voltage, 75–95.
2 " " cobaltic oxide	Amperage, 125–175.
2 " " carbon	Time, 1½ minutes.

The crucible was lined with magnesia. The alloy was very hard, brittle, and feebly magnetic, with a specific gravity of 7.14. Its analysis gave 54.57 per cent. of molybdenum, and 45.35 per cent. of cobalt.

Experiment V.—

4 grams of molybdenum trioxide	Voltage, 70–100.
2 " " cobaltic oxide	Amperage, 110–150.
2 " " carbon	Time, 1½ minutes.

Here I used a graphite crucible, placing the molybdic oxide in the bottom. The alloy was soft enough to be filed, but was brittle and slightly magnetic. The specific gravity was 6.55. It gave, upon analysis, 49.47 per cent. of molybdenum, and 50.86 per cent. of cobalt.

While tungsten and molybdenum ordinarily show many similarities in their reactions, we observe in the preceding experiments differences not wholly devoid of interest. Thus, while tungsten and bismuth did not yield an alloy, with molybdenum and bismuth definite products did result. The experiments with tungsten and copper were positive, but with molybdenum it seemed impossible to alloy copper. Both metals failed to unite with tin, and while this was true of tungsten with manganese, the latter metal and molybdenum combined with apparent readiness. Cobalt, chromium, and nickel, of the seven metals whose oxides were used with the oxides of tungsten and molybdenum, seemed to alloy with the greatest ease with the tungsten and

molybdenum, yielding products which, in the case of cobalt and nickel, may prove to possess a commercial technical value, if prepared in large amounts.

A METHOD FOR THE RAPID DETERMINATION OF CARBON IN STEEL.

By Robert Job and Charles T. Davies.

Received September 25, 1900.

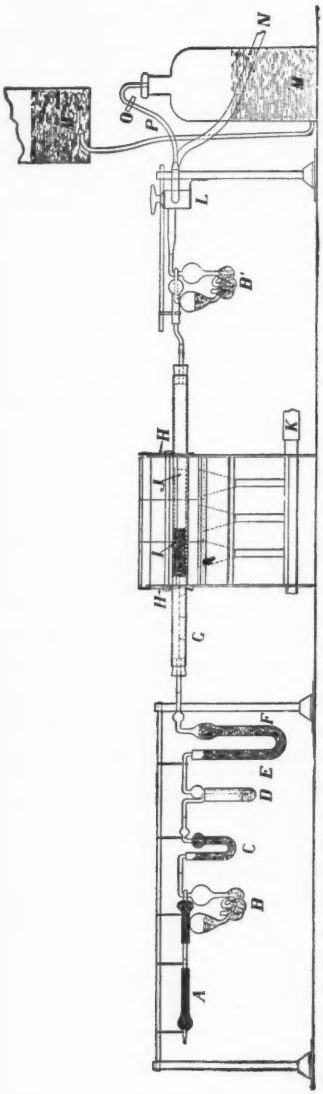
AFTER a very thorough trial of Dr. Sargent's apparatus for the rapid determination of carbon,[1] an investigation was begun with the object of retaining the continuous heating arrangement, and of reducing the apparatus to the simplest possible form without impairing in any way the accuracy of the results.

At the outset it was found that the separate, water-jacketed, copper oxide tube could be entirely eliminated by simply increasing the length of the combustion furnace to 9 inches, increasing the length of the porcelain combustion tube to 20 inches, and inserting closely rolled copper gauze about 4 inches in length, thoroughly oxidized prior to use, into the combustion tube, exactly as in the old method, placing pieces of clay-pipe stems between the copper oxide and the end of the tube in order to prevent the former from being forced out of place when the boat was run up against it in the determination.

Experiments with the furnace itself showed that three Bunsen burners each about 2¼ inches apart, having spreaders upon the tops, furnished sufficient heat to keep the porcelain tube at a bright red heat over 8 inches of its length. A thin sheet-iron shield was placed just beneath the tube as a protection from the direct action of the flame.

After the removal of the separate copper oxide tube, it was found that the gases, upon reaching the calcium chloride tube preceding the weighed bulbs, had been cooled to the temperature of the room, so that the condensing worm was no longer necessary, and further test after its removal proved that the excess of water remaining in the gas at that point was small, and was readily removed by the calcium chloride without necessity of frequent changing. It was further proved that the absorption

[1] This Journal, **22**, 277.



of carbon dioxide in the bulbs was complete under these conditions, and that the temperature of the solution in the latter was not perceptibly raised, even upon very rapid running,—and also that moisture did not escape from the weighed potash bulbs and attached calcium chloride tube, even when the air was run at the rate of 8 bubbles per second, or faster than necessary to complete the combustion in twenty minutes.

Attention was next directed to the sand tube for the removal of chlorine and hydrochloric acid. Under certain conditions, and especially in very warm weather, it was found that the absorption by moist sand was incomplete, and thus that uniformly correct results could not be depended upon with this arrangement. Careful trial was therefore made of various absorbents both wet and dry, and we finally adopted the anhydrous cupric sulphate and cuprous chloride U-tube recommended by Blair, inserting beyond it a small bubble tube containing about 10 cc. of a saturated solution of silver sulphate in sulphuric acid of 1.40 specific gravity which served the double purpose of retaining any hydrochloric acid which might pass the U-tube, and of remedy-

ing the excessive dehydration produced by the copper salts.

The U-tube, E,F, is of the ordinary 6 inch form, and when filled contains cupric sulphate in the arm F, and cuprous chloride in E, both salts being freshly and thoroughly dehydrated, the copper sulphate being nearly white, and the cuprous chloride a dull brown. A small piece of glass wool is placed at the bottom of the tube to separate the salts, and also upon the top. The materials should be in granular form about $\frac{1}{8}$ inch in diameter, free from powder.

After once filling the tube it may be safely used without change until the copper sulphate on the top of the arm F becomes slightly blue or green, or until traces of hydrochloric acid begin to be caught in the silver sulphate solution. The tube should then be removed, placed upon the steam table, or otherwise heated, and a slow suction applied until the cupric sulphate has become nearly white. A second U-tube is kept on hand ready for use and is inserted while the other is being dehydrated. Heating and aspirating for an hour will generally produce the proper dehydration, and the tube is then capped and hung up ready for use. As a general thing a large number of determinations may be made without changing the tube.

The silver sulphate bubble tube, D, is about $4\frac{3}{4}$ inches in height, and $\frac{7}{8}$ inch in diameter, holding about 25 cc., if full. When in use the tube contains from 5 to 10 cc. of the silver sulphate solution, this being blown in from a small wash-bottle. The tube is refilled after a determination if any precipitate has formed. This, however, seldom occurs unless the copper salts have become partially hydrated.

The calcium chloride tube, C, next to the silver sulphate tube retains the excess of moisture from the latter, and the gas passes into the potash bulbs in precisely the same degree of dryness as it emerges from the calcium chloride tube attached to the weighed bulbs, as shown by the fact that in the blank determinations there is almost invariably neither loss nor gain in the weight of the bulbs.

In filling the calcium chloride tubes care should be taken that the material be freshly dehydrated, and in granular pieces about $\frac{1}{8}$ inch in diameter and free from dust. The tubes should be filled as completely as possible, tapping lightly upon them and

filling close to the top, placing a very small piece of cotton at each end.

The potash bulbs, B and B′, are practically the same as those recommended by Dr. Sargent, and hold, when filled for use, about 35 cc. of potassium hydroxide and weigh, including the calcium chloride, about 80 grams. These bulbs may be safely used until they have absorbed 2.5 grams carbon dioxide after which they should be cleaned out and refilled with fresh potash solution of 1.27 specific gravity. The calcium chloride tube, which is about $2\frac{3}{4}$ inches in length, and $\frac{1}{2}$ inch in diameter, should also be refilled with dehydrated material at the same time. The calcium chloride tube, C, immediately before the weighed bulbs, should be refilled in the same manner as soon as the material begins to appear slightly moist at the end nearest the silver sulphate tube. This will usually occur after about fifty determinations. The old calcium chloride when removed can be thoroughly dehydrated in a few minutes by heating in an iron or porcelain dish.

In the course of our work more or less difficulty was at first experienced owing to the cracking of the porcelain tube, especially so when the boats were not warmed before inserting. We found, however, that this could be completely remedied by putting into the combustion tube a piece of platinum foil bent into tubular form, and fitting the combustion tube closely, being careful to have the edges somewhat beveled so that the boat, when pushed in with the wire, would run up smoothly upon the foil, the object of the latter, being, of course, to withdraw the heat absorbed by the boat from a considerable area of the combustion tube instead of from a few points. The above was noted by Dr. Sargent in the paper above referred to.

Attention was next turned to the purity of the oxygen supply, in order to avoid the necessity for the preheating furnace. Investigation showed that commercial oxygen free from hydrocarbons could be readily obtained. Therefore, we merely stipulate with the order that the material shall be of this quality, and upon arrival of the tank run a blank determination, passing the oxygen for ten minutes and then the air in the usual manner. The final weight of the bulbs after forty minutes in the balance should not vary more than 0.2 of a milligram from the original weight. In case, however, a pure supply could not be obtained,

thus necessitating preheating of the gas, we found that a separate appliance could be avoided by simply passing a copper tube through the combustion furnace, utilizing the heat of the 3 burners for the purpose. An apparatus of this nature was attached readily to the furnace by boring two quarter-inch holes in each end of the furnace just above, and slightly to one side of the spreaders of the Bunsen burners, running a seamless copper tube of $\frac{1}{4}$ inch outside diameter entirely through the furnace, out through the end and back upon the opposite side parallel to the first, having the tube in such position that the flame would not play directly upon it, but still so close that the heat would bring it to redness. The ends of this tube, after passing through the furnace, were bent down almost to the level of the desk, and then extended along to the connection of the oxygen supply beyond the three-way cock, L. With this length of about 18 inches from the furnace, the ends of the copper remained cool under all conditions of service, and could thus be attached direct to the rubber tubes leading respectively to the oxygen and to the three-way cock, without any necessity for a water-cooling arrangement.

In the regular determinations we heat up the furnace by lighting the middle burner, turning up one-half for five minutes, then turning up full, at the same time turning up the two end burners one-half, and after five minutes giving the full flame. The tube will be red hot within fifteen minutes from the start, and ready for the day's determinations. If then a boat is in the tube it is pulled out with the wire hook quickly upon a porcelain tile, the weighed potash bulbs, after removing the capillary tips, are connected in position, and the oxygen started through the purifying bulbs, B′, at the rate of about 4 bubbles per second. The determination boat is then removed from the oven, placed within the end of the tube and run quickly into position against the copper oxide with the wire, which is removed at once, and the gum stopper inserted. The oxygen is passed for seven minutes at the above rate, then shut off, and the three-way cock turned and air passed at the rate of about 6 or 7 bubbles per second for twelve and one-half minutes regulating the pressure as necessary by means of the clamp O, and passing about 1 liter of air. The stopper is then removed from the end of the tube,

the bulbs disconnected, and another set of previously weighed bulbs inserted and the second combustion proceeded with. Three weighed bulbs are used in running a set of determinations, and one of the same size but slightly lighter than the others is kept upon the opposite pan of the balances as a counterpoise. The weights are taken after letting the bulbs stand for forty minutes in the balances. The furnace remains hot throughout the day's work without attention.

As to accuracy of results with this apparatus, we have checked repeatedly upon various standard steels, and have obtained uniform and practically identical results with those obtained by the regular combustion method with a platinum tube.

In its present form the apparatus can be readily made by cutting down an old furnace; no water connections are required, the number of joints is reduced to a minimum, the cost of operation as well as the initial cost are greatly reduced below the cost of the old combustion method, a porcelain tube is as satisfactory and quick as a platinum tube, and the entire apparatus from end to end can be placed upon a table 5 feet long.

In this apparatus we have retained what seemed to be for our purpose the most desirable features of the various appliances now in use and wish to make due acknowledgment for them.[1]

We have found it very advantageous to rechlorinate the double chloride of copper and potassium solution after Dr. Sargent's method.[2] As the solution gradually becomes neutral we find it desirable after rechlorinating to make addition of hydrochloric acid in sufficient amount to restore the original acidity, thus preventing the separation of salts, and increasing the rapidity of solution.

The acidity can be determined very easily by titrating 5 cc. of the rechlorinated solution with standard potassium hydroxide solution, taking as the end-reaction the point at which the ferric hydroxide just formed fails to go into solution after shaking. Titration is then made of 5 cc. of a solution of one part hydrochloric acid and 13 parts water (the normal acidity of the double chloride solution) with a drop of neutral ferric salt as indicator, the end-point being determined as above; comparison of the

[1] The potash bulbs and silver sulphate tube were made to order by Queen & Co.

[2] This Journal, **22**, 210.

acidity of the two solutions with the volume of the solution rechlorinated will show the amount of hydrochloric acid needed.

LABORATORY OF THE PHILADELPHIA AND READING
RAILWAY COMPANY, READING, PA.,
September 13, 1900.

DETERMINATION OF IRON IN MAGNETITE ORE BY THE SPECIFIC GRAVITY TEST.[1]

BY JOSEPH W. RICHARDS.

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MANY magnetite ores are simply mixtures of magnetite and quartz. The dressing of such ores at the mines up to a salable percentage of iron is often an important matter, and in such cases the weight of the ore, as determined by its "feel," is the determining factor as to whether it should pass into the ore pile or to the dump. Every new streak of ore opened up brings new material to be thus classified. For such classification, the specific gravity of the ore, carefully taken on a good pair of scales, is of quite sufficient accuracy to answer all practical purposes. Indeed, in many cases, considering the difficulty of obtaining a small representative sample from a mixed lot of ore such as the chemist requires, the specific gravity test made on a considerable weight of sample pieces may be quite as satisfactory as an analysis. The various ways in which specific gravity may be practically determined on an ordinary good scale need not be discussed here; anywhere from 1 to 50 pounds of ore may be tested, according to the scale at hand, and the accuracy desired, and results usually obtained accurate to 0.10 in any case, and often to 0.02 or 0.03, in the specific gravity. Such variations would mean a variation of 1 to 3 per cent. on the iron content, according to the richness of the ore. With ores over 45 per cent. of iron, the variation or error need not be over 2 per cent. on the iron, in any case.

To facilitate such tests, I have calculated the following table of the specific gravity of mixtures of magnetite and silica, of specific gravity of 5.18, and 2.66 respectively, for every 1 per cent. of iron contained, giving in each case also the percentages of magnetite and quartz in the mixture, the latter datum being of particular importance in valuing the ore.

[1] Read at the October meeting of the Lehigh Valley Section of the American Chemical Society.

Fe. Per cent.	Fe_3O_4. Per cent.	SiO_2. Per cent.	Specific gravity.	Fe. Per cent.	Fe_3O_4. Per cent.	SiO_2. Per cent.	Specific gravity.
0	0.0	100.0	2.66	37	51.0	49.0	3.54
1	1.4	98.6	2.67	38	52.4	47.6	3.57
2	2.8	97.2	2.69	39	53.8	46.2	3.60
3	4.1	95.9	2.71	40	55.2	44.8	3.64
4	5.5	94.5	2.73	41	56.6	43.4	3.67
5	6.9	93.1	2.75	42	58.0	42.0	3.70
6	8.3	91.7	2.77	43	59.4	40.6	3.74
7	9.7	90.3	2.79	44	60.8	39.2	3.77
8	11.0	89.0	2.81	45	62.1	37.9	3.81
9	12.4	87.6	2.83	46	63.5	36.5	3.85
10	13.8	86.2	2.85	47	64.9	35.1	3.89
11	15.2	84.8	2.87	48	66.3	33.7	3.93
12	16.6	83.4	2.89	49	67.7	32.3	3.97
13	18.0	82.0	2.92	50	69.1	30.9	4.01
14	19.3	80.7	2.94	51	70.5	29.5	4.05
15	20.7	79.3	2.96	52	71.8	28.2	4.09
16	22.1	77.9	2.98	53	73.2	26.8	4.13
17	23.5	76.5	3.00	54	74.6	25.4	4.17
18	24.8	75.2	3.03	55	76.0	24.0	4.22
19	26.2	73.8	3.05	56	77.4	22.6	4.26
20	27.6	72.4	3.07	57	78.8	21.2	4.31
21	29.0	71.0	3.09	58	80.1	19.9	4.36
22	30.4	69.6	3.12	59	81.5	18.5	4.41
23	31.8	68.2	3.14	60	82.9	17.1	4.46
24	33.2	66.8	3.17	61	84.2	15.8	4.51
25	34.5	65.5	3.20	62	85.6	14.4	4.56
26	35.9	64.1	3.22	63	87.0	13.0	4.61
27	37.3	62.7	3.25	64	88.4	11.6	4.66
28	38.7	61.3	3.27	65	89.8	10.2	4.72
29	40.0	60.0	3.30	66	91.2	9.8	4.78
30	41.4	58.5	3.33	67	92.6	7.4	4.84
31	42.8	57.2	3.36	68	94.0	6.0	4.90
32	44.2	55.8	3.39	69	95.3	4.7	4.96
33	45.6	54.4	3.42	70	96.7	3.4	5.02
34	47.0	53.0	3.45	71	98.0	2.0	5.09
35	48.3	51.7	3.48	72	99.4	0.6	5.16
36	49.7	50.3	3.51	72.4	100.0	0.0	5.18

LEHIGH UNIVERSITY,
October 18, 1900.

IRREGULAR DISTRIBUTION OF SULPHUR IN PIG IRON.

BY RANDOLPH BOLLING.

Received October 8, 1900.

SULPHUR determinations sometimes show marked differences when samples are drilled from different points on the same pig. M. J. Moore[1] has noticed the low results obtained from

[1] This Journal, **21**, 972–975.

"shot samples" as compared with those cast in sand. He states that the results obtained from the "shot samples" are low as compared with the gravimetric determinations. Moore used the volumetric method on his sand and shot samples.

The variations between sulphur determinations, made on drillings from the lower surface of the pig, and those made on the upper surface have been frequently noticed here, and in some cases have been so marked that a cast which was drilled from the top surface of the sample pig, on analysis, showed 0.075 per cent. sulphur, and consequently graded as "off basic" (sulphur running over 0.05 per cent.) was found to be "basic" when the drillings were taken by drilling a hole entirely through the pig; the second determination showed 0.045 per cent. sulphur.

To study the irregular distribution of sulphur a rod 12 × 14 inches was cast in sand by collecting a pound or so at intervals from the runner as a cast was being made until about 10 pounds had been ladled out, and then poured into the mold. This was then drilled at regular intervals of $1\frac{5}{8}$ inches; in all 8 holes were drilled. Sulphur was determined by the cadmium chloride method. The figure represents a cross-section of the rod reduced to a small scale. Beginning at bottom of the rod at the point marked A, the decimal points will indicate where the drill entered. The percentages of sulphur it will be noticed are lower at the bottom of the mold than at the top.

.036
.036
.036
.036
.036
.032
.03
.023
A

It will be seen that the difference between the upper and lower surface amounts to 0.013 per cent., and it would therefore be wrong to report a determination made on drillings taken either at the top or bottom; and to obtain a representative sample it would be necessary to drill a hole entirely through the sample pig, and mix the drillings well before making the determination. By this procedure a fairly average sample would be obtained.

LABORATORY VIRGINIA IRON, COAL, AND COKE CO., BUENA VISTA, VA.

[CONTRIBUTION FROM THE CHEMICAL DIVISION, U. S. DEPARTMENT OF AGRICULTURE, No. 39. SENT BY H. W. WILEY.]

THE COMPOSITION AND ANALYSIS OF LONDON PURPLE.

BY J. K. HAYWOOD.

Received October 22, 1900.

LONDON purple has, up to the present time, been supposed to consist mainly of calcium arsenite and an organic dye residue. From the work which will appear later on in this article, however, it is shown that the arsenic in London purple is principally in the "ic" condition, so that it consists mainly of calcium arsenate, calcium arsenite, and an organic dye residue. On account of the method of manufacture, as I have mentioned in an earlier paper,[1] some of the arsenic will nearly, or quite, always appear in the free condition. It will thus be seen that an analysis of London purple would include the determination of moisture, total arsenic acid, total arsenious acid, soluble arsenic acid, soluble arsenious acid, calcium, and sand.

DETERMINATION OF MOISTURE.

The moisture of London purple, was determined by drying at 100° for from twelve to fifteen hours. On four samples (A, B, C, and D) examined by me the results were :

	A.	B.	C.	D.
	Per cent.	Per cent.	Per cent.	Per cent.
Moisture	1.87	4.07	2.76	2.73

DETERMINATION OF TOTAL ARSENIC.

As regards the determination of total arsenic, it will at once be seen that on account of the presence of the organic dye-stuff residue, and of the calcium, the same methods cannot be applied as those mentioned in my earlier paper for Paris and Scheele greens.

I therefore tried to apply to London purple the same method for determining total arsenic as is used for determining phosphoric acid in phosphates, namely :

1. About 0.5 gram of the sample was oxidized with fuming nitric acid, the excess removed by evaporation, molybdate

[1] "Adulteration and Analysis of the Arsenical Insecticides." This Journal, **22**, 568.

solution added and the yellow precipitate carefully washed, and afterwards dissolved in ammonia, magnesium mixture added and the arsenious oxide weighed as magnesium pyroarsenate ($Mg_2As_2O_7$). In carrying out this method, however, I found that the fuming nitric acid did not oxidize all of the organic dyestuff and that consequently all of the yellow molybdate precipitate did not come down. As the results obtained showed that the amount of arsenious oxide was about 10 per cent. below that which I subsequently found to be present, the method was abandoned.

2. I next tried to determine the total arsenic by dissolving the purple in hydrochloric acid, precipitating with hydrogen sulphide, washing, dissolving in ammonia, reprecipitating with hydrochloric acid, washing first with water, then, after drying, with carbon disulphide, and finally weighing as arsenious sulphide. The arsenious sulphide obtained, however, was perfectly black, from contamination with organic matter, hence this method was also abandoned.

3. The next method which I tried was as follows:

About 2 grams of the sample were dissolved in hydrochloric acid (1 : 4) and heated to from 60°–70° C. to get all arsenic in solution (not higher for fear of driving off arsenious chloride). The solution was then filtered to a definite volume, an aliquot portion taken for analysis and the arsenic precipitated by hydrogen sulphide at 70° C. The precipitate was separated by filtration, washed, and finally both filter and precipitate oxidized with fuming nitric acid in a small flask. After all sulphur and organic matter appeared to be oxidized, the solution was evaporated to a small volume, filtered, treated with magnesia mixture prepared according to the Austin formula,[1] and then with a little ammonia. After settling a few hours the precipitate was washed with ammonia water (1 cc. ammonia to 100 cc. of water) on a Gooch crucible, dried, and the Gooch crucible placed in a solid platinum crucible where it was ignited by degrees in the ordinary way.

Employing this method, the following results were obtained on the four samples (A, B, C, and D):

[1] *Zischr. anorg. Chem.*, **23**, Heft. 2.

	A. Per cent.	B. Per cent.	C. Per cent.	D. Per cent.
Arsenious oxide.....	35.50	37.48	36.47	35.90

Another sample, X, gave results in triplicate, as follows:

Per cent.
33.53
33.32
33.79

Although this method seemed to give fairly good results, I did not feel satisfied for two reasons, namely:

1. The magnesium ammonium arsenate was always dark-colored, showing that the nitric acid had not oxidized all the organic matter, and that consequently when the precipitate was ignited, there would be a loss of arsenic.

2. From the length of time necessary to precipitate the arsenious sulphide (As_2S_3), it appeared to me that some of it at any rate must be in the "ic" condition, and that consequently a method should be employed which would differentiate between the "ous" and "ic" arsenic.

With these two points in view I worked out a method modeled after the Thorn-Smith method, mentioned in my earlier paper, but quite different in all of the small details. It is as follows:

3. Two grams of London purple were dissolved in about 80 cc. of water, and 20 cc. of hydrochloric acid at a temperature of from 60° to 70°, just as in the method above described, filtered, and washed to a volume of 300 cc. One hundred cc. of this was treated in a 500 cc. flask with sodium bicarbonate in excess. The contents of the flask were then brought to the mark with water, using a few drops of ether to destroy bubbles, and 250 cc. filtered off. To this was added starch solution, and the standard iodine solution, until the blue color appeared. The result was the arsenious oxide as such, in 50 cc. of the original solution.

Again 50 cc. of the original solution (representing 0.33333 gram) was heated to 80° on the water-bath, and then taken off and 50 cc. of hydrochloric acid, and three grams of potassium iodide added. The mixture was allowed to stand for at least fifteen minutes, the "ic" arsenic thus being reduced to "ous" arsenic by the action of the potassium iodide, in the acid solution,

iodine being set free. The solution was then rinsed out in a large beaker, and tenth-normal sodium thiosulphate added, drop by drop, to get rid of the iodine. The end-point here was rather difficult to read on account of the very dark color of the solution, but with a little practice one could determine it very easily, by proceeding as follows:

The sodium thiosulphate was run in a little at a time and occasionally a drop of the solution was added to a drop of starch paste. This would of course give a blue color with the starch, which became fainter and fainter as the iodine was used up. Finally when a drop of the solution only gave the slightest blue color, with the starch, a little starch paste was added directly to the whole solution, and the blue color dissipated with a few drops of thiosulphate. With a little practice one could in this way get the exact end-point every time. The solution was immediately made alkaline with solid sodium carbonate. It was again made slightly acid with hydrochloric acid, taking care that all of the solid particles of the sodium carbonate on the bottom were neutralized by the acid,[1] and finally made alkaline with sodium bicarbonate. Starch paste was now added and tenth-normal iodine until the blue color appeared. This end-point is easily read if the beaker is placed on a white surface between the eye and the light, and iodine run in until a distinct purple color appears. The figure thus obtained gave the total amount of arsenic in the solution as arsenious oxide. Subtracting the first figure from this we have the amount of arsenious oxide corresponding to arsenic oxide in 50 cc. of the original liquid. Working in this way I obtained the following results on the samples (A, B, C, and D).

Sample.	Arsenious oxide as such.	Total arsenious oxide present.	Arsenious oxide corresponding to arsenic oxide.	Arsenic oxide present.
	Per cent.	Per cent.	Per cent.	Per cent.
A	8.16	37.21	29.05	33.77
	8.16	37.21	29.05	33.77
	...	36.78	28.62	33.26

[1] If all of the sodium carbonate is not used up, it will itself act on the standard iodine subsequently to be added.

Sample.	Arsenious oxide as such. Per cent.	Total arsenious oxide present. Per cent.	Arsenious oxide corresponding to arsenic oxide. Per cent.	Arsenic oxide present. Per cent.
B	17.45	39.97	22.67	26.34
	17.16	40.12	22.82	26.50
	....	40.26	22.96	26.66
C	10.47	38.67	28.20	32.76
	10.47	38.81	28.34	32.93
D	6.40	37.07	30.67	35.62
	6.40	36.92	30.52	35.45
	...	37.21	30.81	35.79

It will thus be seen that this method not only allows of a determination of the arsenious oxide and the arsenic oxide but also gives very good duplicate results, which results are higher than those obtained by the previous methods. One would expect this on account of the loss which very likely takes place in the last method when the magnesium ammonium arsenate is burned in the presence of some organic matter, which has not been removed by the fuming nitric acid.

DETERMINATION OF CALCIUM OXIDE.

In order to determine calcium oxide in London purple, a portion was dissolved in hydrochloric acid (an aliquot portion of the 300 cc. used in the determination of arsenious and arsenic oxides above would do), and hydrogen sulphide passed through. The precipitate was well washed, the filtrate evaporated to small bulk and transferred to a 200 cc. flask, when it was treated with ammonia (to precipitate the iron, etc.) and made to the mark. A 100 cc. portion of this was filtered off and treated with ammonium oxalate in the usual way. In this manner the following results were obtained on the samples (A, B, C, and D).

Samples.	Calcium oxide. Per cent.
A	25.09
B	23.59
C	24.55
D	25.03
On a fifth sample, X, triplicate results were ..	23.25 23.25 23.40

DETERMINATION OF SOLUBLE ARSENIOUS OXIDE.

To determine the soluble arsenious oxide, the same methods were used as those described in my previous paper for Paris and Scheele greens.[1] On a fifth sample, X, the results were as follows:

Method 1. Washing on the filter not tried.

Method 2. Extracting 1 gram with 500 cc. of water for a number of days at room temperature, and determining arsenious oxide by standard iodine in an aliquot portion of the filtrate.

Sample.	Time.	Arsenious oxide extracted. Per cent.
X	5 days	7.29
	10 days	7.88
	19 days	7.88

Method 3. Extracting 1 gram with successive portions of water at 50°–60° C.

Time.	Arsenious oxide extracted. Per cent.
1 day	7.78
2 days	7.78 + 1.65 = 9.43
3 days	9.43 + 0.87 = 10.30
4 days	10.30 + 0.49 = 10.79
5 days	10.79 + 0.39 = 11.18
6 days	11.18 + 0.29 = 11.47
7 days	11.47 + 0.19 = 11.66
8 days	11.66 + 0.24 = 11.90
9 days	Still gaining.

It will be seen from this that the only method which gave constant results was Method 2, above, just as in the case of Paris and Scheele greens. Applying this method to Samples A, B, C, and D, the following results were obtained:

Sample Nos.	Soluble arsenious oxide. Per cent.
A	2.43
B	13.3
	13.60
C	3.88
D	1.44

DETERMINATION OF SOLUBLE ARSENIC OXIDE.

To determine the soluble arsenic oxide, an aliquot portion of

[1] "Adulteration and Analysis of the Arsenical Insecticides." This Journal, **22**, 568.

the liquid from Method 2 above, for soluble arsenious oxide (say 200 cc.), was transferred to a flask, made slightly alkaline with NaOH, and evaporated to about 25 cc. on a hot plate. The flask was then removed and allowed to cool to about 80°, and an equal volume of concentrated hydrochloric acid and 3 grams of potassium iodide added. It was allowed to stand fifteen minutes, the iodine set free, exactly used up with tenth-normal thiosulphate (the end-point is easily read without the use of starch), and the solution neutralized with sodium carbonate. It was again made slightly acid with hydrochloric acid, taking care that all lumps of sodium carbonate were acted on, then made alkaline with an excess of sodium bicarbonate, and titrated with iodine, using starch as an indicator. From this figure was subtracted the figure representing the amount of soluble arsenious oxide, and the remainder was calculated as arsenic oxide.

Proceeding in this way I obtained the following results:

Sample.	Soluble arsenic oxide. Per cent.
A	15.81
B	7.12
C	12.56
D	19.56

DETERMINATION OF SOLUBLE CALCIUM OXIDE.

In Method 2 above for soluble arsenious oxide and arsenic oxide, I also determined the per cent. of calcium oxide that had been dissolved by the action of the 500 cc. of water. The results on the samples (A, B, C and D) were as follows:

Sample.	Calcium oxide dissolved by 500 cc. of water. Per cent.
A	7.80
B	6.60
C	7.00
D	10.80

DETERMINATION OF OTHER INORGANIC MATTERS.

The inorganic matter, insoluble in hydrochloric acid, mostly consisting of sand, was determined by dissolving the London purple in hydrochloric acid, filtering, washing, and burning the

filter and contents. The results obtained on the samples (A, B, C and D) were as follows:

Sample.	Sand. Per cent.
A	3.54
B	2.61
C	2.46
D	3.55

The following is a condensed statement of the work done:

Sample.	Water. Per cent.	Sand. Per cent.	Total arsenic[1] calculated as As_2O_3. Precipitating as arsenious sulphide, oxidizing with nitric acid. (3) Per cent.	Total arsenic[1] calculated as As_2O_3. Volumetric iodine method. (4) Per cent.	Total arsenious oxide as such. Per cent.	Total arsenic oxide as such. Per cent.	Total calcium oxide. Per cent.
A	1.87	3.54	35.50	37.07	8.16	33.60	25.09
B	4.07	2.61	37.48	40.12	17.31	26.50	23.59
C	2.76	2.46	36.47	38.74	10.47	32.84	24.55
D	2.73	3.55	35.90	37.07	6.40	35.62	25.03
X	....	....	33.55	....	....	.....	23.30

Sample.	Soluble arsenious oxide. Method 2, extraction with 500 cc. cold water. Per cent.	Soluble arsenious oxide. Method 3, extraction with successive portions of water at 60–70°. Per cent.	Soluble arsenic[1] oxide, extracting with 500 cc. cold water. Per cent.	Soluble calcium oxide. Per cent.
A	2.43	....	15.81	7.80
B	13.49	....	7.12	6.60
C	3.88	....	12.56	7.00
D	1.44	....	19.56	10.80
X	7.88	11.90	....	....

FORM IN WHICH VARIOUS CONSTITUENTS APPEAR.

Knowing the full inorganic composition of the four samples given above, we are now in a position to determine the form in which they appear in London purple. The calculation is very simple. If we subtract the soluble arsenious oxide from the

[1] Methods 1 (precipitating with molybdate) and 2 (weighing as arsenious sulphide) were abandoned as unsatisfactory.

total arsenious oxide as such, the soluble arsenic oxide from the total arsenic oxide as such, and the soluble calcium oxide from the total calcium oxide, we have left the insoluble arsenious oxide, the insoluble arsenic oxide, and the insoluble calcium oxide. If the form in which these are united is the normal calcium arsenate, $Ca_3(AsO_4)_2$, and the normal calcium arsenite, $Ca_3(AsO_3)_2$, then the sum of the amounts of calcium oxide, necessary to unite with all the insoluble arsenious oxide to form calcium arsenite, and all of the insoluble arsenic oxide to form calcium arsenate, should be equal to the insoluble calcium oxide as determined.

We will now take up each of the samples and see if such is the case.

A. Total $As_2O_3 = 8.16$ Total $As_2O_5 = 33.60$ Total $CaO = 25.09$
Soluble $As_2O_3 = 2.43$ Soluble $As_2O_5 = 15.81$ Soluble $CaO = 7.80$
Insoluble $As_2O_3 = 5.73$ Insoluble $As_2O_5 = 17.79$ Insoluble $CaO = 17.29$

$As_2O_3 : 3\,CaO :: 5.73 : x$
$x = 4.86 = CaO$ necessary to form $Ca_3(AsO_3)_2$
$As_2O_5 : 3CaO :: 17.79 : x$
$x = 12.99 = CaO$ necessary to form $Ca_3(AsO_4)_2$
$12.99 + 4.86 = 17.85 = CaO$ theoretically necessary to form $Ca_3(AsO_3)_2$ and $Ca_3(AsO_4)_2$
$17.29 = CaO$ actually found.

B. Total $As_2O_3 = 17.31$ Total $As_2O_5 = 26.50$ Total $CaO = 23.59$
Soluble $As_2O_3 = 13.49$ Soluble $As_2O_5 = 7.12$ Soluble $CaO = 6.60$
Insoluble $As_2O_3 = 3.82$ Insoluble $As_2O_5 = 19.38$ Insoluble $CaO = 16.99$

$As_2O_3 : 3CaO :: 3.82 : x$
$x = 3.28 = CaO$ necessary to form $Ca_3(AsO_3)_2$
$As_2O_5 : 3CaO :: 19.38 : x$
$x = 14.15 = CaO$ necessary to form $Ca_3(AsO_4)_2$
$14.15 + 3.28 = 17.43 = CaO$ theoretically necessary to form $Ca_3(AsO_3)_2$ and $Ca_3(AsO_4)_2$
$16.99 = CaO$ actually found.

C. Total $As_2O_3 = 10.47$ Total $As_2O_5 = 32.84$ Total $CaO = 24.55$
Soluble $As_2O_3 = 3.88$ Soluble $As_2O_5 = 12.56$ Soluble $CaO = 7.00$
Insoluble $As_2O_3 = 6.59$ Insoluble $As_2O_5 = 20.28$ Insoluble $CaO = 17.55$

$As_2O_3 : 3CaO :: 6.59 : x$

$x = 5.59$ = CaO necessary to form $Ca_3(AsO_3)_2$

$As_2O_5 : 3CaO :: 20.28 : x$

$x = 14.81$ = CaO neccessary to form $Ca_3(AsO_4)_2$

$5.59 + 14.81 = 20.40$ = CaO theoretically necessary to form $Ca_3(AsO_3)_2$ and $Ca_3(AsO_4)_2$

17.55 = CaO actually found.

D. Total $As_2O_3 = 6.40$ Total $As_2O_5 = 35.62$ Total CaO = 25.03

Soluble $As_2O_3 = 1.44$ Soluble $As_2O_5 = 19.56$ Soluble CaO = 10.80

Insoluble $As_2O_3 = 4.96$ Insoluble $As_2O_5 = 16.06$ Insoluble CaO = 14.23

$As_2O_3 : 3\ CaO :: 4.96 : x$

$x = 4.21$ = CaO necessary to form $Ca_3(AsO_3)_2$.

$As_2O_5 : 3\ CaO :: 16.06 : x$

$x = 11.73$ = CaO necessary to form $Ca_3(AsO_4)_2$.

$4.21 + 11.73 = 15.94$ = CaO theoretically necessary to form $Ca_3(AsO_3)_2$ and $Ca_3(AsO_4)_2$

14.23 = CaO actually found.

On another sample E the results were as follows:

10.91 = CaO theoretically necessary to form $Ca_3(AsO_3)_2$ and $Ca_3(AsO_4)_2$,

10.21 = CaO actually found.

It will be seen from the above calculations that the amounts of calcium oxide, arsenious oxide, and arsenic oxide are such in Samples A, B, and E as to closely approximate the formulas $Ca_3(AsO_3)_2$ and $Ca_3(AsO_4)_2$. In Samples C and D the amounts of these three substances do not so closely approximate the formulas $Ca_3(AsO_3)_2$ and $Ca_3(AsO_4)_2$ but show there is a tendency to form compounds in which the amount of lime is less in proportion to the arsenious and arsenic oxides as in $CaHAsO_4$ and $Ca_2As_2O_5$.

I am inclined to believe from this work that the lime, arsenious and arsenic oxides in London purple are *usually* combined as the normal calcium arsenite and arsenate, but that sometimes small quantities of calcium arsenite and arsenate are formed in which the lime is less in proportion to the arsenious and arsenic oxides as in $CaHAsO_3$ and $Ca_2As_2O_5$. The factors that determine this are very likely the time of boiling, the original concentration of the liquor, the presence or absence of an excess of lime and the form in which the arsenic is present in the original dye residue.

DETECTION OF FOREIGN COLORING-MATTER IN SPIRITS.

[SECOND METHOD.]

By C. A. Crampton and F. D. Simons.

Received October 29, 1900.

A method for the detection of caramel in spirits and vinegar, based upon the selective absorption of caramel coloring-matter by fullers' earth, was published by us in this Journal.[1] Since that time the method has given very satisfactory results in this laboratory in the examination of samples suspected of being artificially colored, the chief difficulty encountered in its application being the variable quality and color-absorbing power of fullers' earth as found in the market. This difficulty was partially overcome by obtaining a supply of the material, of uniform color-absorbing capacity to the degree best suited to the purpose in hand, and in quantity sufficient for a very great number of tests, so that it will not be necessary to change the standards for a long time to come.

We have lately perfected, however, a much more satisfactory and convenient test, which is based simply and solely upon the insolubility in ether of the coloring-matter of caramel and prune juice, the only foreign coloring materials known by us to be used for the artificial coloring of spirits. The coloring-matter of oak wood, on the other hand (principally flavescin), is soluble in ether, which readily removes it from its solution in water or alcohol when shaken with it.

It was found that one treatment with ether was sufficient to completely remove all coloring-matter soluble therein, a second treatment with a fresh portion of ether giving no color whatever. The test is made by shaking the sample with ether, separating the ethereal from the watery layer, and comparing the color of the latter with the color of the original spirits by means of the tintometer. It is necessary, however, to bring the sample to a standard alcoholic strength, as the ether layer takes up most of the alcohol, and consequently the watery layer containing the ether-insoluble coloring-matter would be more concentrated in the case of a high proof than a low proof spirits, giving variable results. In order to insure uniformity in this respect the sample

[1] This Journal, **21**, 355 (1899).

is evaporated on the water-bath until all the alcohol has been driven off; a known quantity of water-white absolute alcohol is then added, which redissolves the coloring-matter precipitated by the removal of the alcohol, making a bright solution, which is then made up to the original volume, and its color taken by means of the tintometer. A definite portion of this solution is then treated with a definite volume of ether, the watery layer separated, and its color taken by the tintometer. A comparison of the two readings will show the character of the sample, genuine spirits giving up about 40 to 50 per cent. of its color, while spirits colored by caramel alone are entirely unaffected, the ethereal layer remaining water-white.

For making the ether extraction we have found it very convenient to use a separatory funnel, which is a slight modification or rather simplification, of Bromwell's modification of Röse's apparatus for the estimation of fusel oil in spirits.[1] Our apparatus is shown in the accompanying figure, and is simply an ordinary globe-shaped separatory funnel or flask with its stem above the stop-cock expanded into a bulb holding 25 cc., the graduation coming up in the stem. The upper bulb should have a capacity of about 100 cc.

Following is the detailed method of procedure: Fifty cc. of the sample under examination are measured out at a standard (room) temperature, and evaporated on the water-bath nearly to dryness; the residue is washed into a 50 cc. glass stoppered flask, 25 cc. absolute alcohol added, and the solution, after cooling to the standard temperature, is made up to the mark with water. After mixing, 25 cc. of the solution are transferred to the separatory apparatus and treated with 50 cc. ether for half an hour, being shaken at intervals; at the end of this period, the layers having separated well, the lower laver is made up with water to the original volume, 25 cc. This is conveniently accomplished by connecting

[1] Bull. No. 49, Chem. Div., Dept. of Agriculture, p. 115.

the lower end of the apparatus with a rubber siphoning tube which carries water from an elevated flask, the inflow of water being regulated by the stop-cock of the apparatus. The contents of the flask are again shaken, and again allowed to separate (whereby the volume of the lower layer is slightly increased), and the watery layer is drawn off through the stop-cock for a reading of its color in the tintometer. At the same time a reading is taken of the 25 cc. of the solution which was not subjected to the treatment with ether; from these two readings the amount of color extracted by ether is calculated in percentages.

The accuracy of the test is much promoted by adhering carefully to the same conditions throughout, especially with regard to temperature.

Applied to a series of 34 samples of spirits known to be naturally colored, the test gave the following results:

	Color removed. Per cent.
Maximum	51.1
Minimum	36.4
Average	41.7

A series of 17 samples known to be artificially colored gave the following figures:

	Color removed. Per cent.
Maximum	23.2
Minimum	0.0
Average	14.8

The results do not show quite so wide a variation between the two sets of samples as the results obtained from the same samples by the fullers' earth test, but the ether extraction method is more satisfactory in a general way. The results obtained by the two methods were confirmatory in every case, and the former will be valuable as a supplementary test. In comparing the figures it must be kept in mind that the results by the two methods are in opposite directions; that is, fullers' earth removes the color from an artificial spirit, while ether removes the color from a natural spirit.

Since the work on this test was completed our attention was drawn to an article by Leach,[1] wherein he recommends the

[1] This Journal, **22**, 207 (1900).

detection of caramel coloring in milk by means of its insolubility in ether, or rather its separation in this way from other coloring-matters (annatto and aniline orange), its identification by "any of the usual tests" to follow.

We have found the method inapplicable to vinegars colored with caramel, genuine apple vinegar giving no color when shaken with ether.

LABORATORY OFFICE OF INTERNAL REVENUE,
U. S. TREASURY DEPARTMENT, WASHINGTON, D. C.

[CONTRIBUTION FROM THE DEPARTMENT OF FOOD AND DRUG INSPECTION OF THE MASSACHUSETTS STATE BOARD OF HEALTH.]

A RAPID METHOD FOR THE DETECTION OF "ANILINE ORANGE" IN MILK.

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DURING the year ending September 30, 1900, there have been examined by the Department of Food and Drug Inspection of the Massachusetts State Board of Health 6137 samples of milk, of which number 45, or 0.7 per cent., contained foreign coloring-matter. Of these colored samples, 20 contained annatto, 3 contained caramel and 22, or 48 per cent., contained "aniline orange" which generic term is used to describe those azo colors used for this purpose. The statistics of the previous five years show that 0.6 per cent. of the total samples of milk were artificially colored, of which 10 per cent. contained aniline orange. The statistics of 1900 show a decided increase in the use of aniline orange as a milk "improver."

In view of the facts that if a sample is carefully colored with a view of not getting in too much, that many samples containing color are above the legal standard and that it is very difficult to tell at a casual glance whether or not a sample is colored, a rapid method for the detection of foreign coloring-matter in milk would be invaluble to the milk analyst.

In testing a sample of milk for formic aldehyde by the well-known method of boiling with an equal volume of strong hydrochloric acid containing ferric chloride,[1] the writer found upon

[1] See Twenty-ninth Annual Report of the Massachusetts State Board of Health, 1897, p. 558.

mixing the cold acid with the milk that the resulting curd was of an intense pink color. Upon further examination by the method of A. E. Leach[1] the sample was found to contain aniline orange. As a result of this, the following method is offered for the detection of aniline orange in milk.

Place about 15 cc. of milk in a porcelain casserole and add about the same quantity of hydrochloric acid (sp. gr. 1.20) gently shaking the casserole to cause thorough mixing and to break up the curd into rather coarse lumps. If the milk contains aniline orange the curd will be colored pink while if it be free from the color, the curd will be white or yellowish, according to the natural color of the milk.

If it is desired to test the milk for formic aldehyde, this same solution may be boiled after the usual addition of a drop of ferric chloride solution and if it be present the usual purple color will appear. The presence of both substances has no effect upon either test.

Commercial hydrochloric acid to which ferric chloride has been added may be used provided that the solution is not too yellow. It has been the custom in this department to add 5 cc. of a 10 per cent. solution of ferric chloride to 2 liters of commercial hydrochloric acid, and to use this solution in testing for aniline orange and formic aldehyde in milk. Sulphuric acid cannot be employed for this purpose.

This test has been in use in this department during the past year and has given satisfactory result

It is unfortunate that this test does not detect annatto and caramel for the analyst frequently labors in vain upon suspicious looking samples, but by applying this simple method, all the samples colored with aniline orange will be pointed out at the same time that the analyst makes the test for formic aldehyde, without the loss of time, reagent, or sample.

[1] A. E. Leach : This Journal, **20**, 207 (1900).

NOTE.

Preparation of Triphenylchlormethane.—In my paper on "The Preparation of Triphenylchlormethane," in the November number, page 752, this Journal, a misstatement occurs which gives an ambiguous meaning to the paper. The first paragraph should read: "It is generally stated * * * *triphenylmethane* is the principal product of the reaction," and not *triphenylchlormethane*, as it reads now.

I also wish to take this opportunity to state that Thorp ("Inorganic Chemical Preparations," p. 39) recommends the preparation of aluminum chloride, in small quantities, by the same method as given in my paper. M. GOMBERG.

NEW BOOKS.

DIE AETHERISCHEN OELE. Von E. GILDEMEISTER und FR. HOFFMANN, bearbeiten im auftrage der firma Schimmel & Co. in Leipzig. Berlin: verlag von Julius Springer. 1899.

THE VOLATILE OILS. By E. GILDEMEISTER and FR. HOFFMANN. Written under the auspices of the firm of Schimmel & Co., Leipzig. Authorized translation by EDWARD KREMERS, Madison, Wis. Milwaukee: Pharmaceutical Review Publishing Co. 1900.

THE CHEMISTRY OF ESSENTIAL OILS AND ARTIFICIAL PERFUMES. By ERNEST J. PARRY, B.Sc., etc. London: Scott, Greenwood & Co. 1899. New York: D. Van Nostrand Company.

The subject of the essential or volatile oils has until very recently been given a distinctly secondary place in the chemical text-books in comparison with the class of fixed oils. Indeed, a few lines about terpenes and camphors has been thought quite a sufficient treatment by the writers of some of the most reputable text-books until quite lately. But this state of things could not remain in view of the brilliant work done in recent years in this branch by men like Wallach, Tiemann, and von Baeyer. As Prof. Emil Fischer, of Berlin, has recently remarked, it is a chapter of organic chemistry that has undergone more rapid development within the past fifteen years than any other.

It is fortunate that, the need having been felt, it should have been filled by the firm of Schimmel & Co., of Leipzig, who are so intimately and honorably connected with the industry of the

essential oils, stepping forward and commissioning scholarly and competent men like Drs. Gildemeister and Hoffmann to write a book covering this ground. The result is the magnificent volume before us in which, as Prof. Kremers says, we find a happy blending of history with chemical science and technology that is quite unique in modern chemical literature.

With true German thoroughness, the work begins with an Historical Introduction, in which we have an account of the spice trade in antiquity and during the middle ages, illustrated by two colored charts, showing "the highways of commerce" in ancient times and in the middle ages, respectively.

This introduction also contains a history of volatile oils, and a history of the methods of distillation and of distilling apparatus. This portion of the work shows much antiquarian knowledge and research and is from the pen of Dr. Fr. Hoffmann, the former editor of the "Pharmaceutische Rundschau," of New York, now resident in Berlin. It is copiously provided with bibliographic references and is of very great interest to the historical student.

The General Part which follows, discusses first the theoretical basis for obtaining volatile oils by steam distillation and then takes up for enumeration and scientific description the more commonly occurring constituents of volatile oils. This descriptive part is very valuable to the chemist, as it epitomizes the latest and most accurate results in this group, and gives full references to the journal literature in foot-notes. This is followed by an account of the methods of examination of volatile oils, both physical and chemical, and lastly we have a list of plants arranged according to families, from which volatile oils are obtained.

The third and longest division of the book is the Special Part, in which we have the history, origin, preparation, properties, composition, examination, and commercial statistics of the volatile oils. The number of individual oils so treated in detail in the original German work is 422, and in Prof. Kremers' translation 430.

Besides the two colored charts already referred to as found in the Historical Introduction, we have two colored maps of Ceylon and of Calabria and Sicily, respectively, and numerous wood-

cuts illustrating the conditions of production of some of the essential oils.

The German publication forms a stately volume of 919 pages, bound in half morocco, and will form a most satisfactory addition to any chemical library.

A few words specially with regard to the American edition. In Prof. Kremers, of the University of Wisconsin, the authors found a most competent translator and editor for the English issue, as it is a field in which he has himself made valuable contributions and with which he is entirely familiar. By condensing several of the sections in the Historical Introduction and putting the bibliographical notes together at the end of the book he has been able to bring all the matter in the compass of 733 pages, without sacrifice of any important feature.

The charts and illustrations are all reproduced and the book is well printed and finely bound.

The work of Mr. Parry on "The Chemistry of Essential Oils and Artificial Perfumes," which also appeared in 1899, while not so ambitious a work as that of Drs. Gildemeister and Hoffmann is, nevertheless, a valuable work, written throughout in a scientific spirit and with a careful consideration of available sources of information. Without attempting anything in the way of a history of the subject, it starts in with the general properties of the essential oils briefly stated, and then gives an excellent review of the compounds occurring in essential oils, stating the most approved views with the aid of structural formulas. The sections on the preparation and analysis of the essential oils are relatively brief and deal with the subject only in a general way. The systematic study of the oils, which takes up the body of the book, deals with them according to the botanical relationship of the plants yielding them, that is, according to natural orders. This part seems to have been well done and the journal literature is considered throughout. A chapter on terpeneless oils follows. The chemistry of artificial perfumes, to which 41 pages are devoted, is well treated, in fact is the best summary of our knowledge which, as far as the writer knows, has appeared in the English language. It makes frequent reference to the patent literature, giving numbers and dates of patents, giving an account of the ionone and violet-oil patent litigation, quoting Tiemann's

and Fritsche & Co.'s patents in full, explains the methods for the manufacture of vanillin, heliotropin, artificial musk, hyacinth, etc. The appendix contains a valuable table of the constants for the more important essential oils. The book can be thoroughly commended as furnishing a compact and convenient reference book on the subject. SAMUEL P. SADTLER.

CHEMISCH-TECHNISCHE UNTERSUCHUNGSMETHODEN. Herausgegeben von DR. GEORG LUNGE. Dritter Band, mit 104 abbildungen. Vierte auflage. xxii + 1082 pp. Berlin: Verlag von Julius Springer. 1900. Price, M. 23.

This volume contains Mineral Oils and Examination of Lubricants, by Dr. D. Holde; Oils and Fats and Special Methods of the Oil and Fat Industry, by Dr. Rob. Henriques; Examination of Resins, Balsams and Gum Resins, and Drugs and Galenical Preparations, by Dr. Karl Dieterich; Caoutchouc and Caoutchouc Wares, by Dr. Rob. Henriques; Ethereal Oils, by Dr. E. Gildemeister; Raw Materials, Products, and By-products of Sugar Manufacture, by Dr. O. von Lippmann and Dr. Georg Pulvermacher; Starch, by Dr. C. von Eckenbrecher; Spirits, by Dr. A. Ebertz; Brandy and Liqueurs and Vinegar, by Dr. G. Schüle; Examination of Wine, by Dr. Karl Windisch; Beer, by Prof. L. Aubry; Examination of Vegetable Material Containing Tannin, by Dr. C. Councler; Leather, by Dr. Joh. Paessler; Paper, by W. Herzberg; Ink, by O. Schluttig and Dr. G. S. Newmann; Organic Preparations, by Dr. J. Messner; The Tartaric Acid Industry and Citric Acid Manufacture, by Dr. Hermann Rasch; and Organic Dyes and the Examination of Textile Fibers and Loading Materials, by Dr. R. Gnehm.

This is a wide range of subjects, which seem in the main to be well covered. The authors confine themselves very closely to methods of testing. Very little is said about methods of manufacture. This seems on the whole something of a defect, for while it is true that information of this sort is otherwise attainable, it is widely scattered and one of the chief values of such a compilation as this is found in the saving of the time of the busy working chemist. Very few chemists can expect to fully cover, even in analytical knowledge, so wide a range as is here presented.

The "Anhang" contains numerous tables from the body of the work printed on one side of the paper for pasting on the

laboratory wall. Some of these tables are: Correction of the Specific Gravity of Petroleum for Different Temperatures; Specific Gravity of Aqueous Glycerine Solutions; Refractive Index of Aqueous Glycerine Solutions; Specific Gravity of Methyl Alcohol; Specific Gravity of Mixtures of Ortho- and Paratoluidin; Determination of Monoethyl and Diethyl Anilin; Solidifying Points of Acetic Acid; Specific Gravity of Oxalic Acid Solutions.

The book is a monument of patient industry and is worth more than it costs. It is a good piece of book work mechanically considered. E. H.

BOOKS RECEIVED.

Bulletin of the United States Geological Survey, No. 176. Some Principles and Methods of Rock Analysis. 114 pp.

The Hessian Fly in West Virginia and How to Prevent Losses from its Ravages. By A. D. Hopkins, Ph.D. Bulletin 67. W. Va. Agricultural Experiment Station, Morgantown, W. Va. August, 1900. 16 pp.

Report on the Geology of the Essequibo, Potaro, Konawaruk and Demerara Rivers, British Guiana. Georgetown, Demerara. 1900. 82 pp. 4to.

Quantitative Chemical Analysis, adapted for use in the Laboratories of Colleges and Schools. By Frank Clowes, D.Sc. (Lond.), and J. Bernard Coleman, A. R. C. Sc. (Dublin). Fifth Edition. Philadelphia: P. Blakiston's Son & Co. 1900. xxiv + 592 pp. Price $3.50.

Decision of the U. S. Circuit Court of Appeals on the Indoine Blue Case. 27 pp. Kuttroff, Pickhardt & Co., P. O. Box 2885, New York City.

Chemisch-technische Untersuchungsmethoden. Herausgegeben von Dr. Georg Lunge. Dritter Band, mit 104 in den Text gedruckten Abbildungen. Vierte vollständig umgearbeitete und vermehrte Auflage. Berlin: Verlag von Julius Springer. 1900. xxii + 1082 pp. Anhang iv+55. Price, 23 marks.

Victor Von Richter's Textbook of Inorganic Chemistry, edited by Prof. H. Klinger, University of Königsberg. Authorized Translation by Edgar F. Smith, Professor of Chemistry in the University of Pennsylvania. Assisted by Walter T. Taggart, Instructor in Chemistry. Fifth American from the Tenth German Edition, carefully revised and corrected. With 68 illustrations in wood and colored lithographic plate of spectra. Philadelphia: P. Blakiston's Son & Co. 1900. xii + 430 pp. Price, $1.75.

An Investigation into the Causes of So-called Uric-acid Lesions,

and a Rational Therapeusis of the Uratic Diathesis. By Alfred Careno Croftan, A.M., M.D., Pasadena, Cal. Reprinted from the New York Medical Journal, for September 22, 1900. 22 pp.

The Economic Entomology of the Sugar Beet. Bulletin No. 60. Agricultural Experiment Station, Urbana, Ill. August, 1900. 136 pp.

Commercial Organic Analysis. A Treatise on the Properties, Proximate Analytical Examination, and Modes of Assaying the Various Organic Chemicals and Products Employed in the Arts, Manufactures, Medicines, etc., with Concise Methods for the Detection and Determination of Their Impurities, Adulterations, and Products of Decomposition. By Alfred H. Allen, F.I.C., F.C.S. Third Edition, rewritten and enlarged. Vol. III, Part I. Tannins, Dyes and Coloring-matters, Writing Inks. Revised and edited by J. Merritt Mathews, Ph.D. Philadelphia: P. Blakiston's Son & Co. 1900. 589 pp. Price, $4.50.

A Text-book of Important Minerals and Rocks, with Tables for the Determination of Minerals. By S. E. Tillman. New York: John Wiley & Sons. 1900. ix + 176 pp. Price, $2.00.

A Handbook of Industrial Organic Chemistry Adapted for the Use of Manufacturers, Chemists, and all Interested in the Utilization of Organic Materials in the Industrial Arts. By Samuel P. Sadtler, Ph.D., F.C.S. Third Revised and Enlarged Edition. Philadelphia: J. B. Lippincott & Co. 1900. Price, $5.00.

Chemical Technology or Chemistry in its Applications to Arts and Manufactures. Edited by Charles Edward Groves, F.R.S., and William Thorp, B.Sc., with which is Incorporated Richardson and Watts' Chemical Technology. Vol. III.—Gas Lighting. By Charles Hunt. Illustrated. Philadelphia: P. Blakiston's Son & Co. 1900. Price, $3.50.

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Parker, C. Leroy, Patent Office, Washington, D. C.
Peet, B. W., Ypsilanti, Mich.
Pemberton, Ralph, 1947 Locust St., Philadelphia.
Phalen, Wm. C., School of Mines, Socorro, N. M.
Phelps, Isaac King, Kent Chem. Lab., New Haven, Conn.
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CHANGES OF ADDRESS.

Blair, A. W., Expt. Sta., Lake City, Fla.
Cushman, Allerton S., Bryn Mawr College, Bryn Mawr, Pa.
Davis, W. W., care Va. Iron, Coal, and Coke Co., Bristol, Tenn.
Goldstein, Harris, 228 Henry St., N. Y. City.
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Lorenz, H. W., Box 4028, Philadelphia, Pa.
Smith, E. A., Republic, Wash.
Wesson, David, care Southern Cotton Oil Co., Savannah, Ga.

ADDRESS WANTED.

Schoen, Joseph, formerly of 3602 Lake Ave., Chicago, Ill.
Wood, J. R., formerly of 439-441 Green St., Philadelphia, Pa.

MEETINGS OF THE SECTIONS.

WASHINGTON SECTION.

The regular meeting was held October 12, 1899.

The first paper was read by Mr. J. K. Haywood and was entitled "The Determination of Glycogen," by J. K. Haywood and W. D. Bigelow.

The authors proved that methods for the estimation of glycogen, which depend on its direct inversion into dextrose, are unreliable and have modified the method of Brücke so as to make it accurate and fairly rapid.

The second paper was read by Dr. F. K. Cameron and was entitled "A Method for Estimating Black Alkali in Soils."

The method enables the determination of the degree of hydrolization of the sodium carbonate in soils and soil crusts containing this compound. It was shown that an accurate determination of the amount of sodium carbonate could not be made by titrating directly with a standard acid, two reactions taking place with the formation of the acid carbonate in varying quantities and furthermore the probable existence of acid carbonate in the soil adds to the difficulty of such a determination. It was shown that acid potassium sulphate is free from these objections, the reaction taking place with quantitative exactness according to this equation :

$$Na_2CO_3 + HKSO_4 = NaHCO_3 + NaKSO_4$$

both substances indicated in the right-hand member of the equation being natural. It should be borne in mind that acid sodium carbonate although a neutral substance towards indicators, quite rapidly and readily inverts with the formation of the alkaline normal carbonate, so that a reasonable degree of speed must be used in making the titrations.

Details of the practical application of the method with examples from practice, were given, and it was shown that in ordinary practice the method was easily capable of an accuracy indicated by a probable error of less than 0.02 per cent.

The last paper was read by Dr. H. W. Wiley, and was entitled "The Fifteenth Annual Meeting of the Association of Official Agricultural Chemists, at San Francisco, July 5–7, 1899."

Mr. Tassin exhibited a specimen of calcium chloride which he

had obtained from a muck soil found in Utah. The salt occurs as an incrustation between Salt Lake City and Salt Lake.

Dr. Bolton exhibited a bibliography of thallium compiled by Miss Martha Dunn and recently published by the Smithsonian Institute. He called attention to the work done in Paris by Jules Garcon, who has published a bibliography of the "Chemical Technology of Textile Fibers," and a pamphlet entitled "Resources of Bibliography of Chemistry." The latter consists of a list of chemical bibliographies.

The regular meeting was held on November 9, 1899.

The first paper of the evening was read by Dr. H. Carrington Bolton and was entitled "Reminiscences of Bunsen and the Heidelberg Laboratory, 1863-65."

The speaker related anecdotes of Bunsen, and events that occurred in the Heidelberg Laboratory during the third semester which he spent there. Contemporary with him were the following Americans:

Eli W. Blake, afterwards professor of physics, Brown University, deceased; Owen W. Root, afterwards professor of chemistry, at Hamilton College, N. Y., deceased; Charles Wolf, of Cincinnati, deceased; Frank Slingluff, of Baltimore; Harry McBurney, of Boston; Lyman Nichols, of Boston; Arnold Hague, of the U. S. Geological Survey.

The second paper of the evening was read by Dr. H. Carrington Bolton and was entitled "Chapters on the History of the Thermometer; I.—The Open Air Thermoscope of Galileo."

The primitive form of the thermometer was invented about the year 1595 by Galileo; this is proved by extant letters addressed to him by his pupil and friend Sagredo. The instrument was an open air thermoscope of the inverted type, and was early applied to meteorological observations, to testing the temperature of fever patients, and to noting temperature of freezing-mixtures.

The very common statement that the thermometer was the invention of C. Drebbel, of Holland, has no basis of fact, as shown by his own publications, copies of which were exhibited by the speaker.